TECHNICAL MEMORANDUM MAA-66-20

TITANIUM-6Al-4V LOW DENSITY INCLUSION PROBLEM

W. P. CONRARDY

MATERIALS APPLICATIONS DIVISION

JULY 1966

RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*

2. REPORT DATE
July 1966

3. REPORT TYPE AND DATES COVERED
Tech Memo/Final July 1966

4. TITLE AND SUBTITLE
TITANIUM-6Al-4V LOW DENSITY INCLUSION PROBLEM

5. FUNDING NUMBERS

6. AUTHOR(S)
W. P. CONRARDY

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Materials Applications Div
Air Force Materials Laboratory
Wright-Patterson AFB, OH 45433

Ref: Thomas D. Cooper, WL/MLS

8. PERFORMING ORGANIZATION REPORT NUMBER
TM-MAA-66-20

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for Public Release; Distribution is Unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*
This memorandum reviews a current materials problem area with respect to undesirable low density inclusions being produced in Ti-6Al-4V alloy. The potential catastrophic effect of these defects is indicated, possible sources of the contamination discussed, limitations with respect to available NDT techniques described and recommendations made for further work on the problem.

14. SUBJECT TERMS

15. NUMBER OF PAGES
16

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT
Unclassified

18. SECURITY CLASSIFICATION OF THIS PAGE
Unclassified

19. SECURITY CLASSIFICATION OF ABSTRACT
Unclassified

20. LIMITATION OF ABSTRACT
Unclassified

ABSTRACT

This memorandum reviews a current materials problem area with respect to undesirable low density inclusions being produced in Ti-6Al-4V alloy. The potential catastrophic effect of these defects is indicated, possible sources of the contamination discussed, limitations with respect to available NDT techniques described and recommendations made for further work on the problem.

PUBLICATION REVIEW

This report has been reviewed and is approved.

William Postelnek

WILLIAM POSTELNEK, Lt Col USAF
Chief, Materials Applications Div.
Air Force Materials Laboratory

RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

TECHNICAL MEMORANDUM
MAA TM 66-20
JULY 1966

MATERIALS APPLICATIONS DIVISION
AIR FORCE MATERIALS LABORATORY
RESEARCH AND TECHNOLOGY DIVISION

TITANIUM 6Al-4V LOW DENSITY INCLUSIONS

I. PURPOSE:

To report on the investigation made by the Air Force Materials Laboratory with respect to low density inclusions in titanium alloys.

II. FACTUAL DATA:

1. Early this year it was brought to the attention of AFML engineers that USAF contractors fabricating hardware components from titanium alloys had encountered a serious materials problem. From as early as 1961 foreign matter, variously referred to as "contamination", "alpha segregation" or "low density inclusions" had been observed in finished hardware as well as in billet and raw forging stock of Ti-6Al-4V alloy. Early work to identify the nature of these observed defects indicated that they were being introduced during the melting process. There is also reason to suspect that this contamination can exist in other titanium alloys. (There are no published accounts of this problem in the literature.)

2. Investigation into the history of these occurrences gave considerable cause for concern. Positive identification of these defects as the primary cause of failure in a number of gas turbine engines (commercial and military) was established. This was manifested by the

cracking and rupture of Ti-6Al-4V compressor discs (forgings) on aircraft and on test stands or spin pits (See Figs 1, 2, and 3). Failure from this cause was also reported in relatively thin wall missile case material. The anticipated increase in titanium usage for both rotating engine components and advanced airframe structural uses precipitated this investigation.

3. The low density inclusions observed have often consisted of voids surrounded by hard (Rc 50), brittle, stabilized alpha grains. (Fig 4) In addition to the relatively massive type inclusions shown in the figures, of greater concern are the smaller ones which probably occur more frequently and are more difficult to detect. It has not been established that all of these defects spring from the same source. The aluminum content in these areas has varied from slightly below to considerably above specification requirements whereas the vanadium has been generally below the requirement. In each instance the oxygen and/or nitrogen contents (as accurately as can be determined) have exceeded permissible specification values.

4. There are several possibilities as to the origin of these defects. In order that the reader may fully appreciate the potential sources of contamination during the production of titanium alloys, a brief review of the process is in order. Titanium tetrachloride - the result of processing rutile ore with chlorine - is reduced to titanium "sponge" in small pellets of convenient size by reduction with either sodium or magnesium. This reaction occurs at elevated temperature and the reaction product must be protected from air and other contamination during the reaction and subsequent cooling to room temperature. The sponge is chipped from

the reaction vessels, crushed, and iron contamination removed with magnets. The material is then acid leached to remove the sodium or magnesium chloride, rinsed, sized, and dried in ovens. All of these processes must avoid the introduction of contaminants, including oxygen and nitrogen which can be picked up in quantity if the material is overheated in air (i. e., during cooling or during drying). This sponge is then thoroughly mixed with crushed or chopped "master alloy" (60% aluminum - 40% vanadium) and a quantity of pedigreed scrap. There is an increasing awareness in the industry with respect to strict control of scrap. Attempts are being made to keep scrap of individual alloys segregated, clean and uncontaminated with foreign material, and limits are placed on the quantity (25-30%) which is added to the mix. Thus the sponge,master alloy and scrap is thoroughly mixed, compressed into briquettes and then welded into a large electrode shape for the initial melt. Double arc melting is a standard practice in the industry,the ingot obtained from the first melt serving as the electrode for the second melt (no additional welding required). During this procedure the electrode and melt must be protected by vacuum or inert gas. The ingot obtained from the second melting is then forged into billets which are sold to prime forgers for shaping into engine and airframe components.

5. The most suspect areas relate to contaminated titanium sponge particles. Discoloration of sponge particles is an indication of high interstitial contamination. Discolored particles have been found to contain up to 19 percent nitrogen. These nitride particles can have melting points as high as 5200°F, approximately 2000°F higher than unalloyed titanium. The nitrided particle, which may not entirely melt

in the arc, is extremely brittle itself and could yield a brittle alpha-enriched zone in the titanium matrix. When forged, the brittle particle or area could fracture leaving an internal discontinuity (Figure 5). A second possible source is oxygen contaminated raw material. The effects of high-oxygen particles would be similar to nitrided particles, in that the particles would be brittle and would yield a high-alpha micro-structure. Other sources are oxygen introduced during melting such as water and air leaks, and contamination during electrode changes, welding or hot topping.

6. Exact procedures and controls on the processes described above differ with the individual suppliers. The methods of initial reduction to titanium metal, kind and amount of inspection for quality in sponge, master alloy and scrap, addition of strengthening material to the briquettes (TiO_2), controls during welding of first melt electrodes (inert atmosphere, dry box, with or without welding wire), chemistry controls throughout, electrode charging and changing and atmosphere control during the double melt are not standardized.

7. The ingot obtained from the second melt must meet the chemistry requirements specified for the alloy being produced. Non-destructive testing of the ingot is primarily to locate and size the "pipe" produced during the melting. The large dimensions of these ingots (about 30" diameter and 10-12 feet long) are such that ultrasonic testing for the small low density inclusions is not practical at this stage.

8. The AFML investigation has disclosed that each of the major titanium producers is aware of the problem and has experienced these kinds of defects in his own production facilities. Since the exact

cause has not been pinpointed, each of the suppliers has made an overall attack on the problem by tighter quality standards including inspection and record keeping with respect to all the raw materials and processes used through the stage of the second melting. Various studies have been and are being conducted in artificially "seeding" electrodes to determine whether the contaminated material would appear in the final ingot. Limited triple melting experiments are included in this work. Some parallel evaluation of nondestructive testing techniques for locating and identifying the defects which were implanted is being conducted.

9. Early in the development of titanium alloys a common contaminant was that of heavy metals (high density inclusions) such as tungsten and molybdenum. These are relatively easy to locate using radiographic NDT techniques. This is still being used in the industry on raw materials and some finished parts such as blading for gas turbines. The low density inclusions are very difficult to observe with radiography. A considerably better NDT technique for low density inclusions is that of immersion ultrasonics. Recently the industry has started using such new equipment for the purpose of finding low density inclusions. However the sensitivity of this method leaves much to be desired particularly on large diameter billet stock which is used as starting material for large forgings. (C5A engine and airframe components require very large billet stock). The present defect standards being used for example are as follows:

3/64" flat bottom hole for bar stock to 1-1/2" thick

5/64" flat bottom hole for billets up to 9" diameter

8/64" flat bottom hole for billets larger than 9" diameter

Enough defects are being found with this equipment and the techniques presently used to make it a matter of genuine concern. It means that small defects cannot be "seen" in increasing diameter material. This is very undesirable for several reasons. A considerable investment in forging and machining a large complex end item may result in its rejection at the final inspection when the initial defect appears on the surface. Though costly, this is currently the best way to find such defects. If the end item does not reveal a surface defect, it may be necessary to over spin test it (rotating engine parts) at maximum use conditions to better insure the absence of potentially catastrophic internal defects. It is infinitely more expensive to locate internal defects through post mortem analysis of aircraft or missile failures.

III. CONCLUSIONS

1. Low density inclusions in titanium alloys can cause catastrophic failures in aerospace equipment.

2. Low density inclusions are still occurring in the production of Ti-6Al-4V alloys. There is no assurance that all of these defects are being detected by available NDT and rejected even in moderate size billet. There is even less assurance as billet sizes increase.

3. There is little hope for significant practical improvement in available NDT techniques for large size billet in the immediate future.

4. The precise cause of low density inclusions is unknown. Coupled with the lack of an adequate NDT system to automatically reject defective material, the situation must continue to be viewed as serious.

5. There is currently insufficient effort on this problem to insure its early resolution.

IV. RECOMMENDATIONS:

1. The Applications Division AFML should alert other services and DOD contractors to this problem.

2. Applicable military specifications should be reviewed and revised as necessary to increase the Quality Control and inspection requirements.

3. Users of titanium for critical rotating components or other structural uses where fatigue is a problem should consider levying specification requirements of the kind described in references 9 and 10.

4. The AFML should increase its support to the ultimate solution of this problem. The following activity is recommended:

a. The Metals and Ceramics Division conduct a study to better characterize the nature and composition of low density inclusions with consideration of compositional controls for its elimination.

b. The Metals and Ceramics Division support a program to (1) improve the sensitivity of current NDT (ultrasonic) techniques for large diameter titanium billets and (2) to explore alternate potentially useful methods for detecting low density inclusions.

c. The Manufacturing Technology Division sponsor a manufacturing and process control study to include sponge production, electrode make-up, artificial seeding and determination of low density inclusions after single, double and triple melts.

d. When defective material availability permits, the Applications Division determine the significance of low density inclusions on the mechanical behavior of this alloy.

e. The Applications Division work closely with the titanium industry, encourage the exchange of new and useful process controls, without disclosing proprietary information; encourage the general use of additional manufacturing controls where these are found to be beneficial to the end product; and levy increased specification requirements as necessary to enforce the controls.

REFERENCES

1. AFML (MAG) letter to the three principle titanium producers dated 25 March 1966 entitled "Alpha Segregation" in Titanium Alloy.

2. R. L. Smith and E. F. Bradley, "Discussion of Segregation in AMS 4928 Titanium-Base Alloy Gas Turbine Engine Components" dated 30 March 1966 P&W Aircraft, East Hartford, Conn.

3. W. P. Conrardy, Memo for the Record, dated 5 April 1966, regarding discussions held at the AFML with representatives of the Oregon Metallurgical Corp on low density inclusions.

4. W. P. Conrardy, Memo for the Record, dated 13 April 1966, regarding visit to Reactive Metals, Inc. plants at Niles and Ashtabula, Ohio on 12 and 13 April 1966.

5. W. P. Conrardy, Memo for the Record, dated 22 April 1966 regarding discussions held at the AFML with a representative of the Titanium Metals Corp of America.

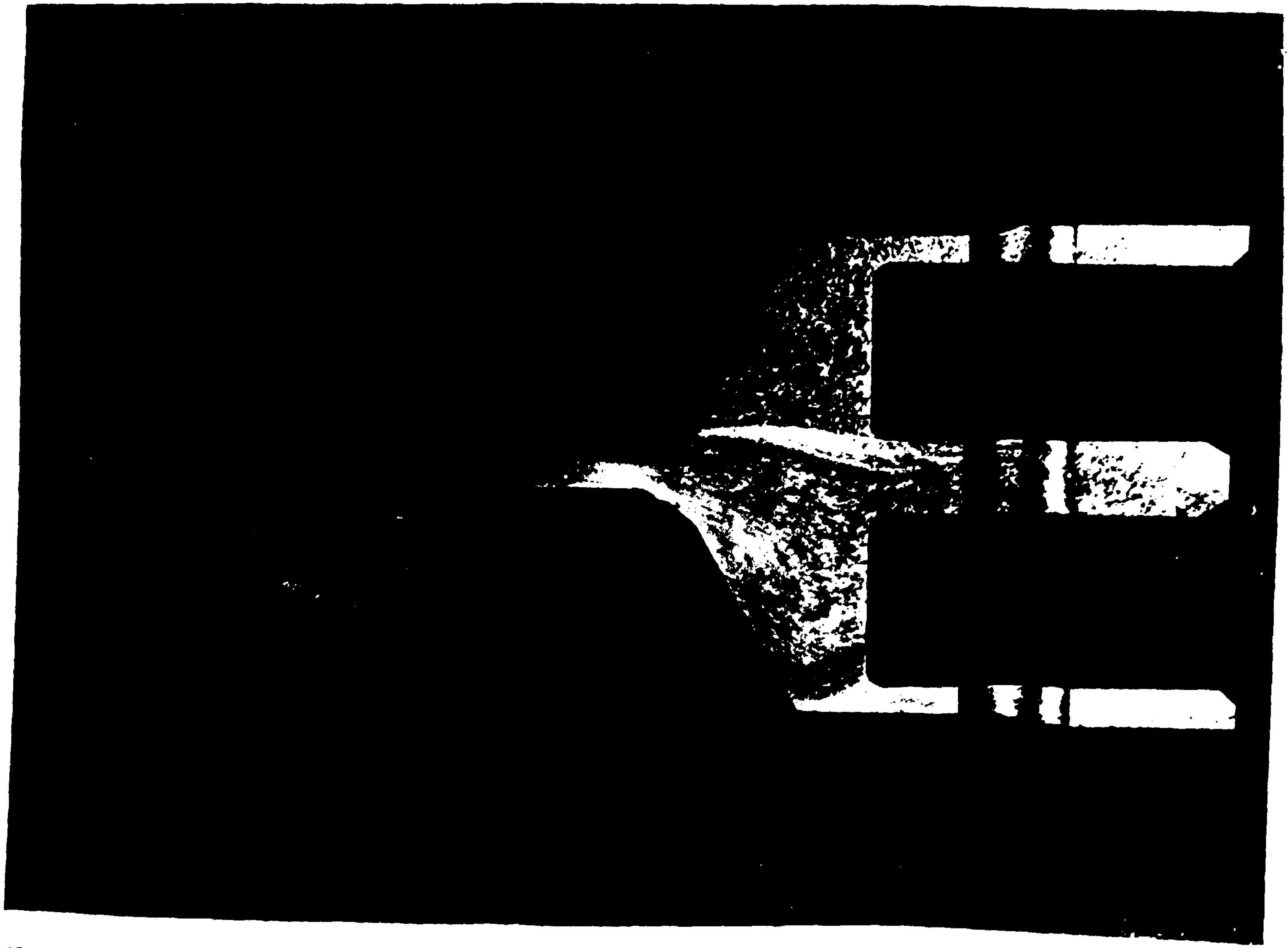
6. Lt Vince Russo, Minutes of 26 April 1966 Meeting with General Electric to discuss Ti-6Al-4V usage in TF-39.

7. W. P. Conrardy, Travel Report on visit to TMCA production facility at Toronto, Ohio on 5 May 1966.

8. Tom Cooper, C. Bradley Ward, Travel Report on visit to TMCA sponge and ingot production facility at Henderson, Nevada on 17 May 1966.

9. P&W Aircraft Div, Materials Control Laboratory Manual Section F-12, Page 1 et ff "Vendor Control of Product Material-Titanium Forgings and Rolled Rings (and Parts Made Therefrom)" revised 1-31-1966.

10. General Electric Co., Evendale, Ohio TF-39 Specification "Titanium Melting and Billet Inspection For Critical Rotating Components".



ETCHANT: KROLL'S FIGURE 1 MAG: 1-1/2X

MACROETCHED RADIAL SECTION OF FAILED COMPRESSOR DISC SHOWING EXTENT OF CONTAMINATED [illegible]

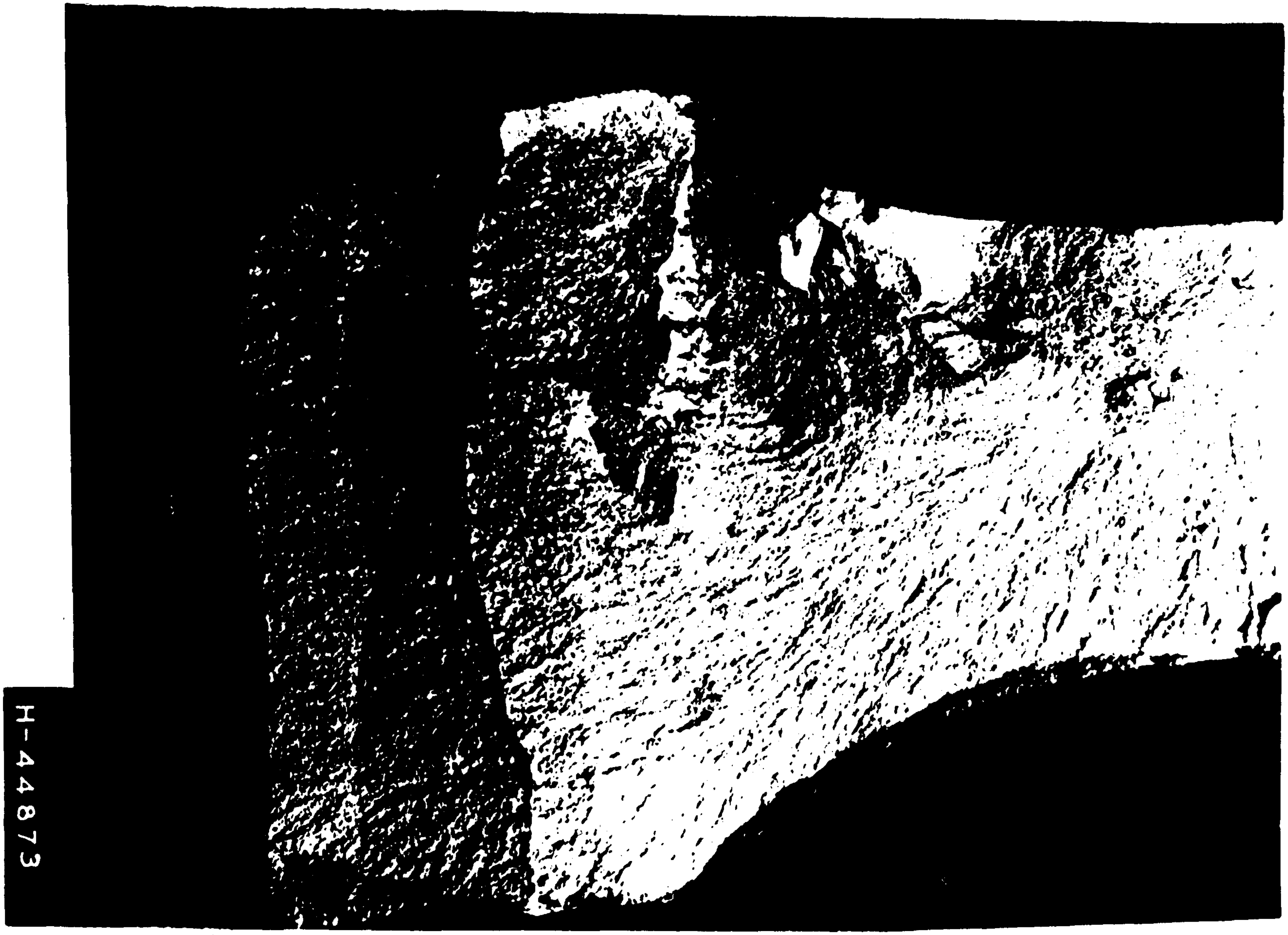
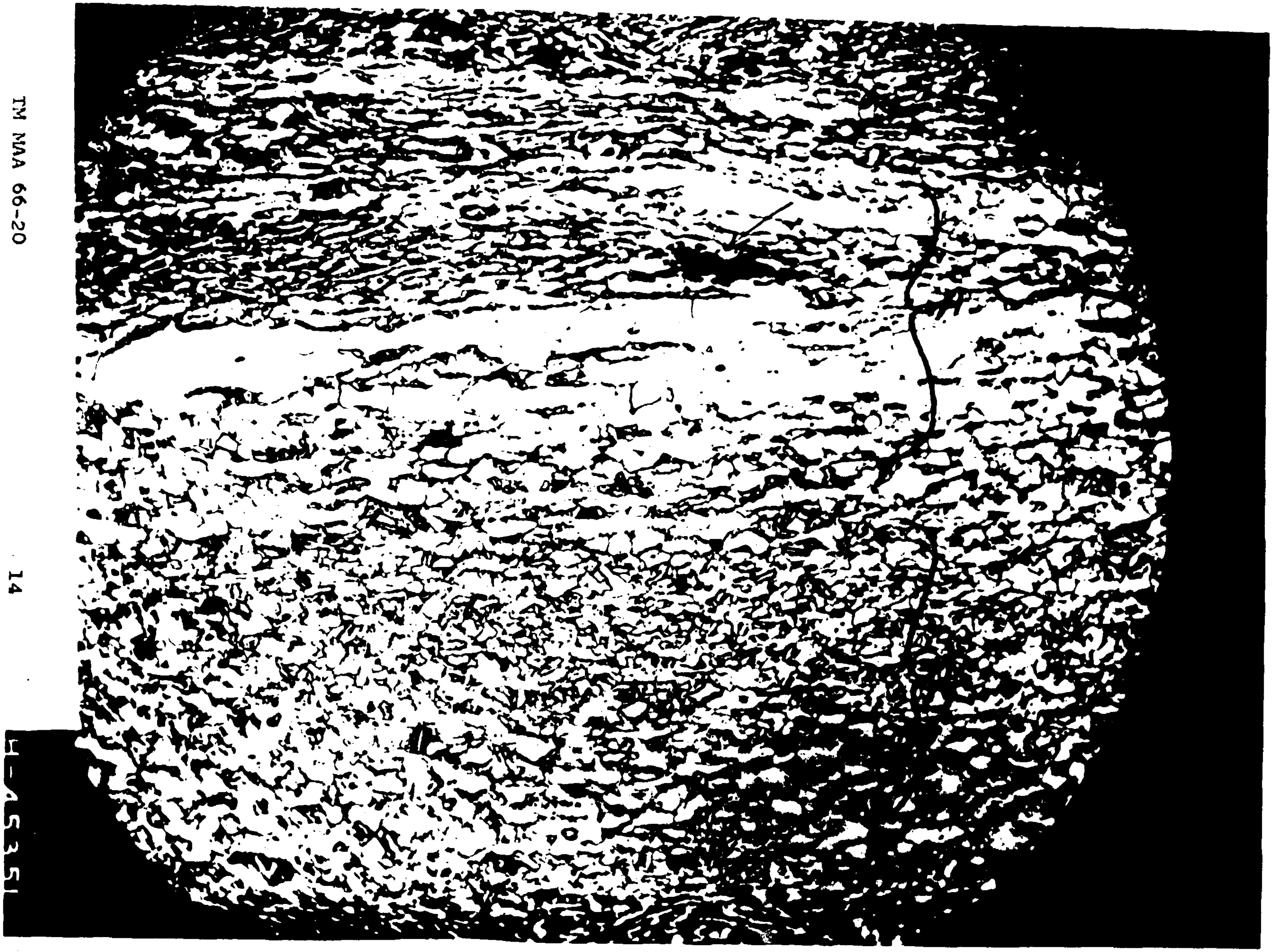


FIGURE 2 MAG: 6-1/2X

SURFACE OF COMPRESSOR HUB FRACTURE SHOWING TENSILE FAILURE PROGRESSING FROM GRANULAR, BRITTLE FRACTURE AREA INDICATIVE OF CONTAMINATION.

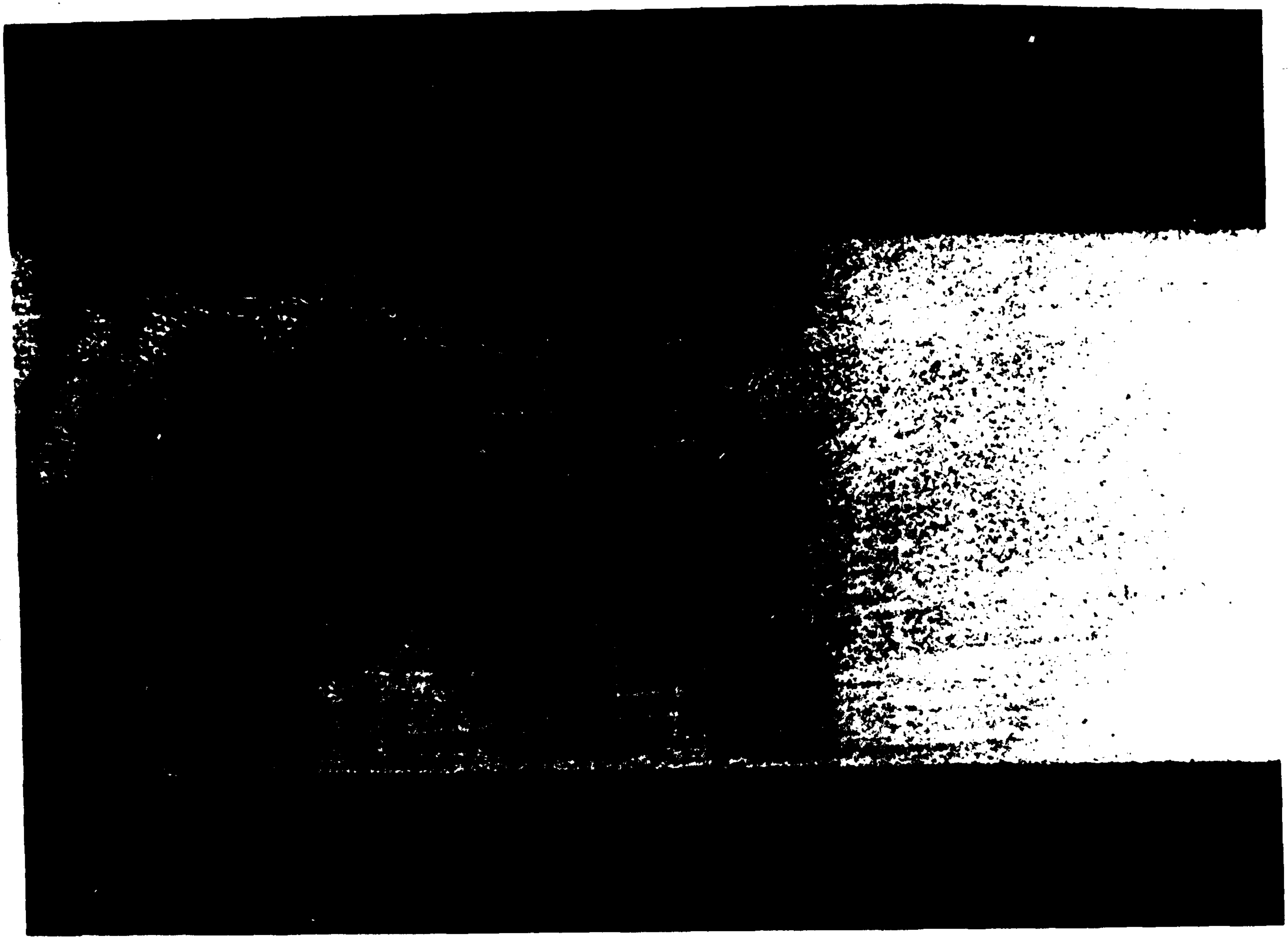
MAG: 5X

SURFACE OF COMPRESSOR HUB FRACTURE SHOWING TENSILE FAILURE PROGRESSING FROM AREA OF FATIGUE AND BRITTLE FRACTURE. A VOID (ARROW) AND CONTAMINATION WERE FOUND AT THE ORIGIN.



ETCHANT: KROLL's REAGENT FIGURE 4 MAG: 100X

PHOTOMICROGRAPH OF SECTION THROUGH CONTAMINATED AREA SHOWING A VOID (ARROW), HARD STABILIZED ALPHA STRUCTURE (BRACKET A) AND SOFT ALPHA STRUCTURE (BRACKET B).



ETCHANT: KROLL'S REAGENT FIGURE 5 MAG: 18X

PHOTOMACROGRAPH SHOWING A VOID AND CONTAMINATED AREA. NOTE FORGING LINES FLOWING AROUND HARD STRUCTURE PRODUCED BY THE CONTAMINATION.